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THE WEATHERING RESISTANCE OF SOME COTTON POLYESTER CORESPUN DUC--ETC(U)

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TECHNICAL NOTE

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THE WEATHERING RESISTANCE OF SOME COTTON/ POLYESTER CORESPUN DUCKS EXPOSED AT INNISFAIL, QUEENSLAND

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A B S T R A C T

Two samples of cotton/polyester corespun duck from a previous trial, four samples representative of current production for the Australian Army, and two experimental samples expected to have improved resistance to weathering were assessed for performance in the tropics, by exposure at the Joint Tropical Research Unit, Innisfail, North Queensland.

Two of the samples had much better resistance to weathering than the others. They had been flame-, rot-, and water-proofed and pigmented to give increases of mass above the mass of the loomstate ducks of about 85% and 65%. The first was loaded with chromic oxide and also contained calcium carbonate in more than sufficient quantity to neutralise any acid formed. The second contained carbon black in addition to a little chromic oxide and also sufficient acid acceptors.

Samples rot-proofed with copper 8-quinolinolate without inorganic copper compounds had better resistance to weathering than those rot-proofed with a basic treatment involving cuprammonium hydroxide, but the pigments present also contributed to the improvement. The modified formulation used for Australian Army ducks including the addition of 1%, by weight, of titanium dioxide (rutile) has only given slight improvement. More rutile is needed together with pigments such as chromic oxide, carbon black and calcium carbonate, and the cuprammonium treatment should be discontinued.

CONTENTS

THE WEATHERING RESISTANCE OF SOME COTTON/POLYESTER COREPSPUN DUCKS EXPOSED AT INNISFAIL, QUEENSLAND

1. INTRODUCTION

This paper is one of a series in which the resistance to weathering of cotton/polyester corespun ducks is discussed. In each paper the results of a weathering trial of commercially prepared ducks or yarns are considered. In the first paper (1) on the exposure of both cotton and cotton/polyester ducks, rot- and water-proofed, or rot-, water- and flame-proofed, it was shown that corespun ducks were much less resistant to weathering than the cotton ducks they were designed to replace. In the second paper (2) it was indicated that the resistance to weathering of cloths made from cotton polyester corespun ducks was improved by using an eccentric rather than a concentric spinning process and by using chromic oxide or titanium dioxide (rutile) pigments. A paper on the weathering of unproofed corespun yarns (3) showed that eccentric spinning produced a reduction of about one-tenth of the loss of strength of the conventionally spun yarn of similar linear density.

Eccentric-spun yarn is, however, not yet available commercially and the alternative method of modifying the pigmentation to provide better protection to the polyester core has still to be used. A modified formulation, containing titanium dioxide (rutile) which had previously been shown (4,5) to increase resistance to weathering had been used in proofing corespun ducks for the Australian Army. Four samples, P3-P6, representative of the four weights of duck used by the Australian Army were obtained having the above treatment and were exposed* at J.T.R.U., Innisfail, Queensland. At the same time two of the samples used in the first trial (1) were re-exposed (P1 & P2). P2 was rot-proofed by the previous standard treatment of the firm that manufactured the four army samples. (It had been given the same basic rot-proofing treatment using cuprammonium hydroxide and had somewhat similar pigmentation but without rutile, and P1 was the duck that had the best resistance to weathering in the first trial (a heavily pigmented, rot-, water- and flame-proofed duck).

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* J.T.R.U. Trial No. 106

Two other samples were included, a heavy flame-proofed duck P7 reputed to have very good resistance to weathering and the other P8, a green-coloured cloth, rot-proofed only with copper 8-quinolinolate (copper-8) which is considered to give protection from actinic degradation (5).

Because only commercially prepared samples were used, the scope of this trial was limited but the object was to determine the resistance to weathering of the standard Army ducks compared to other samples, and to ascertain how much improvement can be achieved by varying the rot-proofing and pigmentation.

The cloths were ranked in order of resistance to weathering according to the methods described by Moroney (6).

2. MATERIALS AND TREATMENTS

The materials selected for trial are listed in Table 1, together with details of proofing and construction. Because they were samples from commercial production, the treatments vary in several respects so that a precise determination of the effect of a specific treatment is not possible.

3. EXPOSURE

Specimens (0.4 x 0.5 m) were exposed to the weather at the Joint Tropical Research Unit, Innisfail, hot/wet cleared site on racks facing north, inclined at $17\frac{1}{2}^{\circ}$ to the horizontal for periods of 2, 4, 6, 9, 12, 15, 18 and 24 months. Meteorological data for the exposure period are given in Table 2.

4. EXPERIMENTAL

Unexposed samples and samples withdrawn from weathering were examined at MRL, Maribyrnong. After examination for visible evidence of microbiological growths, waterproofness was determined by a hydrostatic pressure method. The breaking loads of strips 5 x 30 cm were determined, and this material was later used for microscopical and chemical examination.

Proofing materials were removed as described in the Appendix, and the degradation of the polyester cores then estimated by microscopical examination. The mean molecular weight was obtained from viscosity in m-cresol (7). The damage to cotton was assessed by the Congo Red microscopical test (8), and by the determination of copper number (9) and cuprammonium fluidity (10). Pieces of the unexposed samples and those withdrawn after 18 and 24 months were analysed as received and without any proofing removal using standard analytical procedures and the results are given in Table 3.

5. RESULTS AND DISCUSSION

The weathering of corespun ducks can be divided into two parts, deterioration of the polyester and deterioration of the cotton.

5.1 Degradation of Polyester

Most of the strength of corespun cotton/polyester yarns is provided by the polyester core. The degradation of the polyester is indicated in the tests of strength, (Table 4), microscopical assessment, and changes in molecular weight (Table 5), which provided an important clue to the ability of the different treatments applied to the cotton to protect the polyester. The ranking of the cloths in Table 6 from the data of Tables 4 and 5 and from the microscopical assessments of damage not given in detail because of the subjective nature of the test shows that cloth P7 had the best resistance to weathering, P1 was next, then P8 while P2, P3 and P4 had the least resistance to weathering. These latter three were Army ducks.

The ranking according to loss of strength was similar after all periods of exposure but rankings from the results of the other tests varied. It was considered that the best estimate of rank for these tests would be obtained by ranking the totals of the ranks for 9, 12, 18 and 24 months. Rankings of P7, P1, P8 and P6 were similar in the three tests while those for P2, P3 and P4 varied; but the overall ranking was similar. The results for mean molecular weight of the polyester component have been given as percentage decreases on weathering. Cloths P7 and P1 showed decreases of only 7 and 8% after 24 months of weathering compared with 19% for P3. The change was far less than that obtained for loomstate duck in the first trial (1), 65% after 24 months (Table 5). Evidently the pigmented cotton protects the bulk of the polyester but localised damage in certain parts is except for P1 and P7 sufficient to cause considerable reduction in strength. This protecting effect of the rot-proofed cotton was also noted in the microscopical examination of the polyester, damage being much less than that noted on loomstate samples in the earlier trial (1). It was noted that it was very difficult to obtain samples of P7 sufficiently clean for microscopical examination; pigment particles clung tenaciously to the fibres.

From the retained strength shown in Table 4, the cloths may be graded by the exposure times required to reduce the strength to 50% as follows :

P7	20 months
P1	9 months
P8, P6, P5	5 months
P4, P3, P2	3 months

Cloth P7 had the longest life on exposure, more than twice that of the next best. If we compare P7 with P6 which has a similar loomstate mass, the life expected is four times as great.

The resistance to weathering improved with increase of mass per unit area. The effect may be seen by comparing P3-6, samples of similar proofing

and pigmentation. Comparisons made from Table 4 at the level of 30% retained strength, show that the exposures required were 9, 12, 18 and 18 months. The use of 3-fold yarn for sample P5 (Table 1) may have improved the protection of the polyester such that P5 had similar weather resistance to P6 a cloth of greater mass but made from 2-fold warp and single-fold weft yarn.

Cloth P8 was submitted as an alternative to P4. It was rot-proofed with copper-8 alone. Both had similar loomstate masses but the exposures for 30% retained strength were :

P8	Greater than 24 months
P4	12 months

The presence of a larger quantity of copper-8 and the absence of cuprammonium copper in P8 were important factors in the improvement but the different pigments (Table 1) in the two samples and the presence of a green vat dye in P8 may have some effect. All the tests show a large difference in the ranking of P4 and P8 (Table 6).

Although this trial does not provide a direct comparison between rot-proofing with cuprammonium and copper-8, it should be noted that the samples with the best resistance to weathering have all been proofed with copper-8 and those with the worst resistance contain large amounts of copper from the cuprammonium treatment.

5.2 *Degradation of Cotton*

The degradation of the cotton component is measured by microscopical assessment by the Congo red test (Table 7), by copper number (Table 8) and by cuprammonium fluidity (Table 9). The ranking of the cloths in Table 10 was done as described in para. 5.1. The table displays some similarity between the results of the three tests in that P7 shows the best resistance to weathering of the cotton, P1 is next and P8 third. Cloth P2 has the worst resistance to weathering. The effect of the modified formulation used for proofing the Army ducks P3-6 including the addition of 1% of rutile is shown by the better rating for P3 compared to P2. The difference was not as great as that reported previously (2) for an addition of 10% of rutile; at least 3-5% should be used.

As observed in the degradation of the polyester core, cloths having the best resistance to degradation of the cotton had been proofed with copper-8 while those showing the worst resistance had been proofed mainly with cuprammonium. Improving the resistance of the cotton to weathering does not automatically improve the resistance of the polyester but the elimination of the forms of copper such as cuprammonium that accelerate actinic degradation (5) and the inclusion of copper-8 and pigments which retard degradation (5) improves the resistance of both fibres.

The tests for cotton differ in their significance. The Congo red microscopical test (Table 7) shows that the damage to the cotton was entirely actinic; there was no definite evidence of fungal attack or degradation by acid, nor was fungus detected by visual examination of the samples when they

were returned from J.T.R.U. Except for P7, damage to the cotton was from moderate to fairly severe after 2 months. After 24 months, damage was quite severe on all but less on P1 and P7 than on the others.

Copper number determines the reducing power of the cotton cellulose and it increases considerably on weathering particularly in the "non-alkali" samples which had been prepared without any alkaline treatment. The ranking of the results was somewhat similar to the order for the Congo red test in that P1 and P7 were the best and P8 the worst. Samples showing the highest copper numbers after 24 months of exposure had been proofed with cuprammonium.

Cuprammonium fluidity is related to the degree of polymerisation of the cellulose. As the long chain molecules are broken up during weathering, fluidity increases. Although fluidity and copper number measure different properties, the samples are ranked in a somewhat similar order in Table 10 except for P8. The results in Table 9 show that after 18 months, fluidities of all those proofed with cuprammonium were in the 30-35 range while those proofed with copper-8 gave results in range 22-28.

5.3 Water Resistance

Table 11 shows that cloths P2-6 had an initial water resistance of 5-6 kPa and that this was retained or increased during weathering. Cloth P1 lost water resistance slightly on weathering. The results for P7 were variable. The initial value was high but except for the low result after 15 months, water resistance after weathering was almost equivalent to that of P2-6.

Cloth P8 differs from the others; a silicone and aluminium formate waterproofing finish had been applied whereas wax emulsion had been used in all the others (Table 1). Leaching out of the water soluble material may have been responsible for the increase in water resistance during the first 12 months from about 5 to 17 kPa but decomposition of the silicone would be responsible for the subsequent decrease to 2.2 kPa after 18 months. Note the reduction in the silica content, (Table 3).

5.4 Effect of Weathering on Rot-proofing and Pigments

The effects of weathering for 18 and 24 months on the contents of rot-proofers and pigments are shown in Table 3. All samples retained some copper, and fungal attack was negligible. Inorganic copper in samples P2-6 was slowly lost but half to two-thirds remained after 24 months. Samples P3-6 also retained about half of the pentachlorophenyl laurate originally present. Sample P7 lost more pentachlorophenyl laurate but sufficient remained to supplement the fungicidal action of the small amount of copper.

Titanium and chromium used to improve resistance to actinic degradation were not removed by weathering, and antimony in the flame-proofed samples was not lost. The amounts of zinc added as borate for flame-proofing and calcium carbonate were gradually reduced, probably through conversion to the chlorides due to reaction with hydrochloric acid produced by photochemical decomposition of chlorinated compounds. Sample P1 loaded with 10.5% of calcium carbonate lost one fifth while P7 lost zinc rather than calcium.

Sample P8 initially rather acid, (pH of aqueous extract 5 g/100 ml is 4.7), lost most of its calcium. When chlorinated compounds were present in quantity, the amount of calcium lost on weathering indicated that 2-3% of an acid acceptor was required, otherwise 0.2-0.3% sufficed. Some acid acceptor is needed because even loomstate cotton tends to develop acidity during weathering.

Cloth P7 has a smaller inorganic loading than P1 but it also contains carbon black. Only P8 contains silica as a major component; silicone and aluminium waterproofing were used. There was some loss of silica after 18 months showing decomposition or loss of the silicone corresponding with loss of water resistance (Refer Table 11). The amounts of silica in other samples varied with the degree of soiling also shown by an increase in iron and aluminium contents. Soiling was worst on P1 which gained nearly 2% of silica. Iron oxide had been added as a pigment in P2-6 and P8 and was not removed by weathering, the amounts present being fairly constant at about 1.5%.

6. CONCLUSION

The results of this exposure trial have shown that if the samples are arranged in rank according to resistance to weathering, P7 is the best in all tests, P1 is next and P8 is third in most tests. Cloth P2 the standard army duck from the first trial (1) that was re-exposed in this trial was the worst in all tests except the mean molecular weight reduction of the polyester, P3 and P4, army ducks proofed using the modified formulation were next, while P5 is better than or equal to the heavier cloth P6 in all tests except that of the molecular weight of the polyester.

It is believed that inadequate cover of the polyester core is mainly responsible for the deterioration on weathering (1-3). Increasing the mass per unit area of the material has increased the resistance to weathering but the lighter P5 made from 3 fold yarns is often equal or better in resistance to the heavier P6, made from 2-fold and single fold yarns, probably because of the improved cover given by the 3-fold yarns.

The heavy flame-proofed P7 has about four times the life of P6 of similar loomstate mass, and the light flame-proofed P1 has about three times the life of P2 of similar loomstate mass as measured at 50% retained strength (Table 4) but the loading of P1 with chromic oxide and calcium carbonate and P7 with chromic oxide, calcium carbonate and carbon black appears to have been largely responsible. In addition, the three cloths with best resistance to weather have been rot-proofed with copper-8 without using cuprammonium.

The ranking of cotton degradation is similar to that for polyester degradation. The elimination of cuprammonium copper and the use of copper-8 or copper-8 and pentachlorophenyl laurate in larger amounts together with suitable pigments has decreased the degradation of the cotton. The cloths with the best resistance had been flameproofed with compounds of antimony and zinc and chlorinated hydrocarbons but sufficient calcium carbonate was present to neutralise any acid formed during exposure. It has been shown

that 2-3% of an acid acceptor, generally calcium carbonate is required if chlorinated compounds are present, otherwise 0.2-0.3% should suffice. The improved rating for P3 compared with P2 especially for degradation of cotton shows the effect of the modified formulation used for proofing the army ducks. Although several changes were made (Table 1), the improvement imparted by 1% of rutile is comparatively small compared with that given by 10% in previous work (2). About 3-5% should be used to obtain a worthwhile improvement.

7. ACKNOWLEDGEMENTS

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TABLE 1

DETAILS OF PROOFING AND CONSTRUCTION OF THE SAMPLES USED IN THE TRIAL

Sample Cloth		P1	P2	P3	P4	P5	P6	P7	P8
Rot-proofing									
Cuprammonium as basic treatment			x	x	x	x	x		
Copper naphthenate			x	x	x	x	x		
Copper-8		x		x	x	x	x	x	x
Pentachlorophenyl laurate				x	x	x	x	x	
Water-proofing									
Wax emulsion		x	x	x	x	x	x	x	
Silicone and aluminium formate									x
Flame-proofing									
Antimony/zinc/chlorinated hydrocarbons		x						x	
Pigments									(vat dye)
Chromic oxide		x	x	x	x	x	x	x	
Iron oxide			x	x	x	x	x		x
Titanium dioxide (rutile)				x	x	x	x		
Calcium carbonate		x	x	x	x	x	x	x	x
Carbon black								x	
Mass of loomstate fabric, g/m^2		200	200	200	270	340	410	410	270
Mass of finished fabric, g/m^2		375	270	270	365	470	555	650	375
Threads per cm,	Ends	37	37	38	28	22	20	20	26
	Picks	23	25	24	17	14	14	15	17
Fold of yarn,	Warp	2	2	2	2	3	2	2	2
	Weft	2	1	1	1	3	1	1	2
Breaking load, N/5 cm									
	Warp	1510	1750	1900	2630	3030	3520	3160	2290
	Weft	1160	1360	1240	1740	2220	2680	2980	1630
Mean molecular weight of polyester		10200	10500	10600	10600	10800	10600	10700	10500

TABLE 2

SUMMARY OF METEOROLOGICAL DATA, INNISFAIL, AUG. 1971 - JULY 1973

Property	2 months	4 months	6 months	9 months	12 months	15 months	18 months	24 months
Temperature, ^{o}C								
Mean max	28	30	31	30.5	29	28.5	29	28.5
Mean min	17.5	19	20	20.5	19.5	19	19	19.5
Mean	22.5	24.5	25.5	25.5	24	24	24	24
Mean index of relative humidity, %	80	76	76	79	80	80	79	81
Rainfall (total), mm	63	157	1149	3106	3749	3924	4325	7176
Sunshine (total), h	462	1021	1484	1909	2428	3243	4002	4933
Radiation (total), kwh/m^2	301	612	1000	1403	1748	2296	2918	3590

TABLE 3

ANALYSIS OF FINISHES

Material	Ash %	SiO_2 %	TiO_2 %	Cr_2O_3 %	Al_2O_3 %	Fe_2O_3 %	CuO %	Sb_2O_3 %	ZnO %	$CaCO_3$ %	PCPL %
P1 Unexp	18.4	0.4	Nil	7.2	Nil	0.1	0.1	4.8	0.9	10.5	Nil
18 m	23.0	2.4	Nil	7.6	0.8	2.1	0.03	5.3	0.9	8.8	Nil
24 m	21.4	2.2	Nil	7.7	1.8	1.4	0.1	5.4	0.6	8.4	Nil
P2 Unexp	6.2	0.3	Nil	2.1	Nil	1.4	2.1	Nil	Nil	0.2	Nil
18 m	5.2	0.4	Nil	1.8	0.3	1.4	1.4	Nil	Nil	0.1	Nil
24 m	5.8	0.1	Nil	1.9	0.1	1.5	1.2	Nil	Nil	0.1	Nil
P3 Unexp	7.4	0.2	1.1	1.5	0.7	1.5	2.5	Nil	Nil	0.2	0.12
18 m	7.2	0.3	1.0	1.6	0.6	1.6	1.7	Nil	Nil	0.1	0.04
24 m	7.5	0.3	0.9	1.6	0.5	1.6	1.8	Nil	Nil	0.1	0.07
P4 Unexp	7.4	0.2	1.0	1.4	0.2	1.4	2.3	Nil	Nil	0.2	0.14
18 m	6.8	0.7	1.1	1.6	0.9	1.5	1.7	Nil	Nil	0.3	0.06
24 m	6.6	0.3	0.9	1.6	0.4	1.6	1.7	Nil	Nil	0.2	0.08
P5 Unexp	7.4	0.2	1.2	1.7	0.6	1.7	2.2	Nil	Nil	0.3	0.14
18 m	7.1	1.0	1.0	1.7	0.6	1.3	1.8	Nil	Nil	0.3	0.07
24 m	6.9	0.4	0.9	1.7	0.6	1.8	1.6	Nil	Nil	0.3	0.09
P6 Unexp	6.6	0.1	0.9	1.4	0.5	1.5	2.0	Nil	Nil	0.5	0.16
18 m	6.0	0.6	0.9	1.4	0.2	1.6	1.1	Nil	Nil	0.1	0.07
24 m	6.0	0.3	0.8	1.4	0.1	1.5	1.2	Nil	Nil	0.4	0.06
P7 Unexp	6.9	0.1	Neg	0.4	Neg	0.07	0.02	4.0	1.8	2.6	0.73
18 m	7.3	0.3	Neg	0.4	0.9	0.2	0.04	4.2	1.4	2.5	0.34
24 m	6.8	0.4	Neg	0.4	1.1	0.1	0.06	4.2	0.9	2.5	0.21
P8 Unexp	9.4	2.1	0.1	Nil	4.5	1.6	0.4	Nil	0.2	0.8	Nil
18 m	9.2	1.7	0.1	Nil	5.2	1.7	0.1	Nil	0.2	Nil	Nil
24 m	8.8	1.6	0.1	Nil	4.8	1.4	0.2	Nil	0.1	0.1	Nil

PCPL = Compounds of pentachlorophenol calculated as laurate.

TABLE 4

PERCENTAGE OF WARP BREAKING STRENGTH RETAINED

Period of Exposure	P1	P2	P3	P4	P5	P6	P7	P8
2 months weathering	75	57	55	58	62	62	86	67
4 months "	64	42	43	47	53	52	81	55
6 months "	57	30	36	41	48	47	78	47
9 months "	50	25	30	35	42	41	64	42
12 months "	44	20	26	30	38	37	62	38
15 months "	42	18	22	27	34	35	55	35
18 months "	35	13	17	24	30	30	53	31
24 months "	38	12	16	21	27	28	46	32

TABLE 5

MEAN MOLECULAR WEIGHT OF POLYESTER BY VISCOSITY IN META-CRESOL

(Percentage reduction on results for unexposed material)

Period of Exposure	P1	P2	P3	P4	P5	P6	P7	P8	Loomstate (1) duck from first trial
9 months weathering	5	10	18	10	17	8	2	4	-
12 months "	10	10	13	13	17	17	7	5	-
18 months "	9	17	17	15	14	11	8	11	63
24 months "	7	16	19	22	16	12	8	16	65

TABLE 6

RANKS OF INCREASING DEGRADATION OF POLYESTER

Sample	Retained strength	Reduction in mean molecular weight of polyester	Microscopical assessment of actinic degradation	Mean Rank
P1	2	2	2	2
P2	8	5	7	7
P3	7	8	6	7
P4	6	7	8	7
P5	4	6	5	5
P6	5	4	4	4
P7	1	1	1	1
P8	3	3	3	3

TABLE 7

ASSESSMENT OF ACTINIC DAMAGE OF COTTON BY THE CONGO RED TEST

Period of Exposure	P1	P2	P3	P4	P5	P6	P7	P8
0, Unexposed	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
2 months weathering	A3	A3	A4	A3-4	A3	A4	A1	A3-4
4 months "	A4	A4	A4	A4	A4	A4	A4	A4
6 months "	A4	A5	A4	A4	A4	A4	A3	A4
9 months "	A4	A4	A4	A4	A4	A4	A4	A4
12 months "	A4	A5	A4-5	A5	A4-5	A4-5	A4-5	A4
18 months "	A3-4	A5	A5	A5	A4-5	A4	A4	A4
24 months "	A4	A4-5	A4-5	A5	A5	A5	A4	A4-5

A3 = 30-50% loss of strength of the cotton

A4 = 50-75%

A5 = 75-100%

TABLE 8

COPPER NUMBER OF SEPARATED COTTON NON-ALKALI SAMPLES

Period of Exposure	P1	P2	P3	P4	P5	P6	P7	P8
0, Unexposed	0.2	0.2	0.4	0.5	0.4	0.5	0.6	0.5
6 months weathering	0.8	1.3	1.0	0.7	0.5	0.8	0.8	1.0
9 months "	1.2	1.8	1.4	1.0	0.7	0.6	0.9	1.0
12 months "	0.8	1.7	1.3	0.9	0.9	1.5	0.6	1.0
18 months "	1.2	3.7	3.2	2.2	1.8	1.1	1.3	1.6
24 months "	0.7	3.2	2.8	2.1	1.9	1.7	1.0	1.4

TABLE 9

CUPRAMMONIUM FLUIDITY

Period of Exposure	P1	P2	P3	P4	P5	P6	P7	P8
0, Unexposed	6	10	7	12	10	8	7	11
6 months weathering	18	29	23	22	21	20	16	21
9 months "	19	35	31	27	21	20	17	27
12 months "	25	36	29	30	25	33	19	27
18 months "	24	35	32	32	30	31	23	27
24 months "	24	34	34	32	33	32	22	28

T A B L E 10

RANKS OF INCREASING DEGRADATION OF COTTON

Sample	Congo red test	Copper number	Fluidity	Mean rank
P1	1	2	2	2
P2	8	8	8	8
P3	5	7	7	7
P4	7	5	6	6
P5	6	3	4	4.5
P6	4	4	5	4.5
P7	2	1	1	1
P8	3	6	3	3

T A B L E 11

WATER RESISTANCE IN KILOPASCALS

Period of Exposure	P1	P2	P3	P4	P5	P6	P7	P8
0, Unexposed	5.2	5.8	5.9	5.3	5.1	5.9	9.5	5.1
2 months weathering	4.5	5.6	6.0	6.7	4.8	7.1	10.6	8.6
4 months "	5.4	7.4	6.0	7.3	5.5	9.2	12.7	9.3
6 months "	5.0	6.6	7.5	7.4	5.8	9.1	8.2	9.9
9 months "	4.6	6.0	6.1	6.6	5.7	9.3	7.1	12.2
12 months "	4.7	6.2	6.0	7.3	5.7	8.8	5.0	17.2
15 months "	3.6	6.9	6.6	7.6	5.3	10.1	2.6	10.3
18 months "	4.3	5.9	7.0	7.6	6.2	9.3	5.0	2.2
24 months "	4.5	6.9	5.8	7.3	5.4	10.7	5.0	1.8

APPENDIX

PREPARATION OF CELLULOSIC AND POLYESTER MATERIALS

FOR DETERMINATION OF DAMAGE

Examination by chemical or microscopic methods of deteriorated cotton textiles to determine the type and degree of degradation requires that the cotton fibres be as free as possible from finishing and other extraneous materials. Cotton fibres may also have to be separated from other fibres such as polyester in intimate mixture with them before examination. This paper describes the procedures we use to obtain the fibres in suitable condition.

METHOD FOR REMOVAL OF FINISHING MATERIALS FROM COTTON

The method described will remove most wax and most pigments from cotton as well as phosphorus-based flame-resistant and many other finishes. Many resin finishes will not respond to the treatment.

The treatment is based on information supplied privately some years ago by the Shirley Institute for preparation of material for examination by the Congo red test; we have modified the treatment to cope with changes in finishing processes developed over recent years.

An appropriate amount of material unravelled into yarn is extracted in petroleum ether (40-60^{o}C) or diethyl ether for about 3 h with frequent agitation. After rinsing in solvent and allowing to dry, the material is treated successively with 2% (v/v) acetic acid solution at 40^{o}C for 15 min, 1% oxalic acid solution at 80^{o}C for 15 min, and 1% boiling sodium hydroxide solution for 1 h, rinsing in water after each treatment. The material is finally boiled in water for 15 min and dried at a temperature of 90^{o}C, or less. In order to prevent further degradation of the cotton the temperatures specified must not be exceeded but they should be maintained as nearly as possible. The effect of a treatment of this kind on the results of tests for degree of degradation have been discussed in an earlier paper (1). The cotton material is reduced to short lengths either by disintegrating in a mill or by cutting.

For some tests, for example, determination of alkali solubility, it is obvious that an alkaline treatment cannot be used and the alkali boil has to be omitted. In such a case, or where the normal treatment, that is including the alkali boil, does not appear to have removed most extraneous material, we recommend the addition of up to 10 g per l of a surface active agent in the final water boil. At MRL, Ultravon JU400 (Ciba-Geigy) has been used successfully but other agents are also suitable. If a surface active agent is used final washes in hot water followed by rinses in ethanol and acetone are required.

When examining cotton/polyester materials, some of this "non-alkali" sample is used for separation of the components.

SEPARATION OF COTTON COMPONENT

For the determination of the degree of degradation of the cotton component it is essential to remove the polyester without damaging the cotton. The following method is used. A sample is treated four successive times with m-cresol at about 100°C for 10-20 min each, filtering after each treatment through sintered glass (porosity 2). It is rinsed with cold m-cresol followed by ethanol until free from m-cresol then boiled in water and dried as above. This "non-alkali" sample is used for the determination of copper number, alkali solubility and carboxylic acid but a portion is boiled in 1% sodium hydroxide as described above to obtain a sample for determining cuprammonium fluidity, copper number (alkali treated sample) and for microscopical examination.

SEPARATION OF POLYESTER COMPONENT

In order to obtain polyester for the determination of damage, cotton must be removed without damaging the polyester. Cotton is removed by treatment with cuprammonium hydroxide. The sample is shaken for at least 3 h in cuprammonium hydroxide solution of similar concentration to that used for determining fluidity (2) and the solution filtered through a tared sintered glass filter (porosity 2). The residue is again shaken in cuprammonium hydroxide for 0.5 h and filtered again, washed twice with cuprammonia, then ammonia, water, 4M hydrochloric acid, water (6 times), ethanol and acetone, dried in the oven and weighed.

Clean polyester remains.

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1. Hindson, W.R. (1949). The use of chemical tests in determining the type and degree of damage to cotton fibres. MRL Report 177.

2. ——. (1969). Determination of fluidity of cotton and regenerated cellulose fibres in a solution of cuprammonium hydroxide. Australian Standard Specification ASLS1-1969, Standards Association of Australia.

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